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FRACTURE TOUGHNESS OF HYDROGEN-EMBRITTLED
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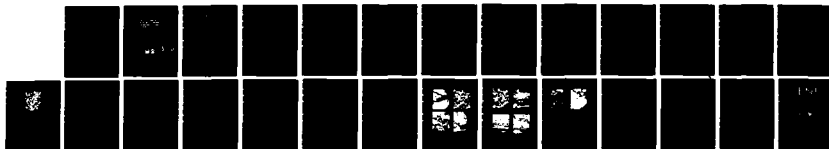
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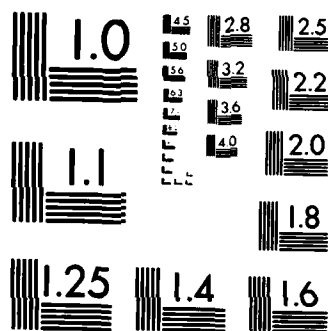
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Fracture Toughness of Hydrogen-Embrittled Precipitation-Hardened Stainless Steels

by
Karen F. Hayes
Engineering Department

APRIL 1985

NAVAL WEAPONS CENTER
CHINA LAKE, CA 93555-6001



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FOREWORD (U)

A study investigating the influence of aggressive environments on the mechanical behavior of precipitation hardenable (PH) stainless steels was authorized by the Naval Air Systems Command under Task Assignment A420401/008-613420000003. The experimental work was performed during the summer of 1982. The purpose of this study was to investigate the mechanism of stress corrosion of PH steels to develop resistance to embrittlement, consistent with strength requirements. This report is one of a series investigating the factors that influence environmental degradation of structural materials used in missile applications.

This report was reviewed for technical accuracy by Dr. G. T. Murray and Dr. G. A. Hayes.

Approved by
D. J. RUSSELL, Head
Engineering Department
1 April 1985

Under authority of
K. A. DICKERSON
Capt., U.S. NAVY
Commander

Released for publication by
B. W. HAYS
Technical Director

NWC Technical Publication 6488

Published by.....Engineering Department
Collation.....Cover, 11 leaves
First Printing.....140 unnumbered copies

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER NWC TP 6488	2. GOVT ACCESSION NO. AD-A156664	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Fracture Toughness Of Hydrogen-Embrittled Precipitation-Hardened Stainless Steels		5. TYPE OF REPORT & PERIOD COVERED Final Report 1982-1983
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Karen F. Hayes		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Weapons Center China Lake, CA 93555		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Task Assignment A420401/008-613420000003
11. CONTROLLING OFFICE NAME AND ADDRESS Naval Weapons Center China Lake, CA 93555		12. REPORT DATE -April 1985
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		13. NUMBER OF PAGES 20
		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) PH stainless steels fracture toughness aggressive environments hydrogen embrittlement stress corrosion fractography metallography		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) See back of form.		

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(U) Fracture Toughness of Hydrogen-Embrittled Precipitation-Hardened Stainless Steels, by Karen F. Hayes. China Lake, Calif., Naval Weapons Center, April 1985, 20 pp. (NWC TP 6488, publication UNCLASSIFIED.)

(U) The chevron-notched specimen method was used to determine the fracture toughness of precipitation-hardened (PH) stainless steels possessing a range of microstructures. The specimens were tested while immersed in various aggressive environments (air, salt water, acidified salt water, or after cathodic polarization).

(U) Chevron-notched test specimens yielded K_{Ic} data which substantiate data obtained by the ASTM E399 test method. It was substantiated that chevron-notched specimens can be used to evaluate K_{Isc} of PH steels tested in aggressive environments. Test results indicated that as yield strength increased, K_{Isc} decreased in all aggressive environments that were evaluated. K_{Isc} consistently decreased as the test environment became more aggressive, with cathodic polarization and cathodic charging leading to the greatest decrease in fracture toughness. This indicated that hydrogen is the primary factor in promoting stress corrosion behavior in the PH steels that were investigated. Fractography showed that as the fracture toughness was decreased, due to aggressive testing environments, the fracture mode changed from dimpled rupture to pure cleavage.

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ACKNOWLEDGMENT

I would like to gratefully acknowledge the technical support of Dr. George T. Murray, professor of Metallurgy, California Polytechnic State University, San Luis Obispo, California. This work was performed at the Naval Weapons Center, China Lake, California during the summer of 1982, with that facility providing financial assistance.

INTRODUCTION

K_{Ic} , the plane-strain critical stress-intensity factor, is ideally measured in an inert atmosphere and represents the inherent ability of a material to withstand a given stress-field intensity at the tip of an existing crack. It has practical significance in that it is an intrinsic material parameter and is recognized as being of great importance, especially in the aerospace and nuclear industries where catastrophic failure cannot be tolerated. Traditional methods of determining fracture toughness are generally recognized as being tedious and expensive. The recently developed^{1,2} chevron-notched specimen for the determination of plane-strain fracture toughness appears to offer a simpler, less expensive alternate test method. This method also appears to have promise for inexpensively determining fracture toughness values of materials subjected to aggressive environments.

This investigation had two objectives. First, the short rod (chevron-notched specimen) test method was evaluated from the standpoint of determining the reliability of measuring critical stress-intensity factors in aggressive environments (K_{Isc}). Second, precipitation-hardened (PH) stainless steels were tested in various environments to provide much needed K_{Isc} data for ongoing ordnance development programs. These results were used to supplement published K_{Isc} data on PH stainless steels, and aided in resolving conflicts in this data.

ENVIRONMENTAL EMBRITTLEMENT OF PH STAINLESS STEELS

Recent work sponsored by the Department of the Navy has been directed toward characterizing the mechanical properties of PH stainless steels exposed to aggressive environments. These studies^{3,4} indicate that significant changes in fracture mode and ductility can be correlated with hydrogen content in 15-5PH³ and 17-4PH⁴ stainless steel, heat treated to a variety of conditions.

¹L. M. Barker. Engineering Fracture Mechanics, Vol. 9, 1977, p. 361.

²L. M. Barker. "Short Rod and Short Bar Fracture Toughness Specimen Geometrics and Test Methods for Metallic Materials," Fracture Mechanics: Thirteenth Conference, American Society for Testing and Materials, ed. by Richard Roberts. ASTM STP 743, 1981, pp. 456-475.

³G. T. Murray. Metallurgical Transactions A, Vol. 12A, (December 1981), pp. 2138-41.

⁴Naval Weapons Center. Hydrogen Embrittlement in 17-4PH Stainless Steel, by K. R. Hayes. China Lake, Calif., NWC, August 1982. (NWC TP 6343, publication UNCLASSIFIED.)

Other investigators^{5,6,7,8,9,10} have also observed varying degrees of stress corrosion cracking (SCC) in PH steels subjected to harsh environments. Fujii⁵ reported that 17-4PH stainless steel was sensitive to SCC at high strength levels, but was relatively insensitive in the overaged condition. The effect of an applied cathodic potential implied the involvement of hydrogen. Significant variation in K_{Isc} with changing cathodic potential was noted. Das⁶ determined the susceptibility of cathodically polarized 15-5PH stainless steel to hydrogen embrittlement (HE). Fracture toughness tests indicated a drop in K_{Isc} with increasing cathodic potentials. Capeletti⁷ showed that the fracture toughness of 17-4PH stainless steel decreased significantly with increased hydrogen gas test pressure for a variety of heat treatment conditions.

THE CHEVRON-NOTCHED SPECIMEN METHOD

Until the late 1970s the plane-strain critical stress-intensity factor, K_{Ic} , of materials was usually measured in air in accordance with ASTM E399. As mentioned earlier, under appropriate conditions this method yields accurate K_{Ic} values of metallic materials; however, it is also a fairly complex and expensive method of determining K_{Ic} . The short-rod method, which utilizes a rod-shaped chevron-notched test specimen, has been developed to test not only brittle materials such as carbides, glass, and ceramics, but also more ductile metals such as alloys of aluminum, titanium, and iron.

⁵C. T. Fujii. "Stress-Corrosion Cracking Properties of 17-4PH Stainless Steel," Stress Corrosion-New Approaches, American Society for Testing and Materials. ASTM STP 610, 1976, pp. 213-25.

⁶K. G. Das, W. G. Smith, and R. W. Finger. Proceedings of Second International Congress on Hydrogen in Metals, Paper 3E1, presented at Paris, France, published by Pergamon Press, Oxford, England, 1978.

⁷T. L. Capeletti. Proceedings of Second International Conference on Mechanical Behavior of Materials, American Society for Metals. Metals Park, Ohio, ASM, 1976, pp. 1489-92.

⁸A. W. Thompson. Metallurgical Transactions A, Vol. 7A, 1976, pp. 315-18.

⁹J. K. Stanley. Journal of Spacecraft, Vol. 9, No. 11, 1972, pp. 796-804.

¹⁰A. W. Thompson, and J. A. Brooks. Metallurgical Transactions A, Vol. 6A, 1975, pp. 1431-42.

The short-rod specimen configuration is shown in Figure 1. With an increasing load, P , applied to the specimen, a crack will eventually initiate at the point of the "V". The load necessary to advance the crack increases until the crack reaches a critical length, a_c ; thereafter, the load decreases with increasing crack length. The peak load, P_c , obtained during the experiment is used to calculate K_{Ic} .

The equation used for determining K_{Ic} is derived from linear elastic fracture mechanics (LEFM) principles, assuming that the energy per unit area of new crack surface created in plane strain is a material constant, G_{Ic} . Therefore, under plane-strain conditions the energy required to advance the crack a small distance, da , is proportional to the material constant, G_{Ic} , and satisfies the equation

$$dW = G_{Ic} b da,$$

where b is the width of the crack front and dW is the incremental work done on the specimen during the test.

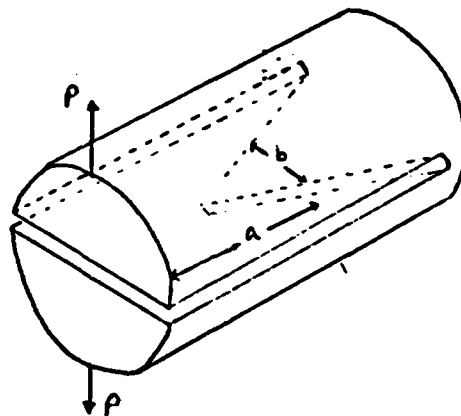


FIGURE 1. Short Rod Specimen Configuration.

Once plastic deformation begins, if the crack is advanced an additional increment, da , an additional load-point-opening increment, dx , will be observed. This new additional irrecoverable work, dW_2 , satisfies

$$dW_2 = \frac{1}{2} P dx.$$

The definition of the elastic compliance of the specimen is $\lambda = x/P$. Letting P be the average value of the load (i.e., a constant) during the crack advance, we have $dx = P d\lambda$. Substituting into the equation above,

$$dW_2 = \frac{1}{2} P^2 d\lambda.$$

According to the Griffith-Irwin-Orowan criterion, dW_2 represents a release of elastic strain energy by the specimen which is available for creation of the new crack area. By equating dW and dW_2 , we obtain

$$G_{Ic} b = \frac{P^2}{2} \frac{d\lambda}{da},$$

where b , P , and $d\lambda/da$ are evaluated at the crack length, a , at which the incremental crack advance took place. The above equation was also published by Irwin and Kies in 1954.¹¹

To obtain the critical stress intensity factor, K_{Ic} , it should be remembered that G_{Ic} and K_{Ic} are related by the equation

$$G_{Ic} = K_{Ic}^2 \frac{1-\nu^2}{E},$$

where ν is Poisson's ratio, and E is Young's modulus. By equating the two G_{Ic} terms,

$$K_{Ic}^2 \frac{(1-\nu^2)}{E} = \frac{P^2}{2b} \left(\frac{d\lambda}{da} \right).$$

¹¹G. W. Irwin and J. E. Kies. "Fracturing and Fracture Dynamics," Welding Journal, Vol. 33, 1954, p. 193.

Simplifying,

$$K_{Ic}^2 = \frac{P^2}{(1-\nu^2)} \cdot \frac{E}{2b} \left(\frac{d\lambda}{da} \right).$$

Taking the square-root of both sides of the equation leaves

$$K_{Ic} = \frac{P^2}{B^{3/2}(1-\nu^2)^{1/2}} \cdot f(a/B),$$

where B is the specimen diameter, and where

$$f(a/B) = \left[\frac{B}{2b} \frac{d(\lambda EB)}{d(a/B)} \right]^{1/2}.$$

The quantity in brackets is only a function of the ratio a/B , independent of the specimen material, as long as the scaled specimen configuration remains constant. As with other fracture toughness geometries, this short-rod geometry can be compliance calibrated, and $f(a/B)$ can be approximated by a polynomial in a/B . Therefore a K_{Ic} measurement can be made advancing a crack to some measured value of "a" (so that $f(a/B)$ can be evaluated), then measuring P necessary to further advance the crack.

Since the quantity $f(a/B)$ is a function only of geometry and is a constant, at the value of the maximum load, P_c , $A = f(a_c/B)$, where A is a constant. Therefore,

$$K_{Ic} = AP_c / [B^{3/2}(1-\nu^2)^{1/2}].$$

If we follow custom by replacing $(1-\nu^2)^{1/2}$ by unity, then

$$K_{Ic} = AP_c / B^{3/2}$$

By replacing the fourth-order polynomial in the last two equations with the dimensionless constant, A, the equations used in determining K_{Ic} values have been greatly simplified. No measurement of crack length is required and the load used in determining K_{Ic} is simply the maximum load obtained during the test. Fatigue pre-cracking of the short-rod specimen is not necessary because a sharp crack is "popped-in" early in the test cycle.

As for limitations of this test method, the short-rod specimen's B-dimension should satisfy

$$B \geq (K_{Ic} / \sigma_{ys})^2,$$

where σ_{ys} is the materials tensile yield strength.¹² By comparison, the traditional LEFM, ASTM E399 test requires that specimens satisfy

$$B \geq 2.5 (K_{Ic} / \sigma_{ys})^2,$$

where B is the specimen thickness, in order that plane-strain conditions prevail.

PROCEDURE

MATERIALS AND SPECIMEN PREPARATION

Fracture toughness specimens were manufactured from vacuum melted 1-inch diameter bar stock from the following materials: 17-4PH stainless steel, PH13-8Mo stainless steel, and 15-5PH stainless steel. The chemical composition of these materials is presented in Table 1 and their mechanical properties are listed in Table 2.

The 17-4PH stainless steel specimens were heat treated to the following conditions: Condition A, H900, H950, H1000, and H1050. The 15-5PH and PH13-8Mo stainless steels were aged to the H1000 condition. Characteristic microstructures of 17-4PH stainless steel in the A, H1000, and H1050 conditions are shown in Figure 2. As expected, the microstructures consisted of martensite for the Condition A sample and tempered martensite for the H1000 and H1050 condition samples.

FRACTURE TOUGHNESS TESTING

Chevron-notched specimens were used to determine the fracture toughness of the PH stainless steel in the presence of various environments. After heat treatment, each specimen was machined in accordance with the short-rod dimensions as developed in Barker.^{1,2} All of the test specimens were 1-inch in diameter. A TerraTek Fractometer® II test system¹² was used for all fracture toughness measurements.

¹²TerraTek, University Research Park. Development of the Fractometer II System for Fracture Toughness Testing Using Short Rod and Short Bar Specimens, by L. M. Barker. Salt Lake City, Utah, TerraTek, June 1979. (TerraTek Report TR 79-32, publication UNCLASSIFIED.)

Table 1. Composition Of PH Stainless Steel Alloys.^a

Alloy	Cr	Ni	Cu	Mn	Cb+Ta	C	P	S	Si	Mo	Al	Fe
17-4PH	16.45	4.67	3.44	.72	.43	.041	.01	.01	.33	-	-	Bal.
15-5PH	15.13	4.71	3.62	.48	.41	.043	.01	.01	.46	-	-	Bal.
PH13-8Mo	12.5	8.2	.02	.01	-	.033	.001	.004	.01	2.14	1.00	Bal.

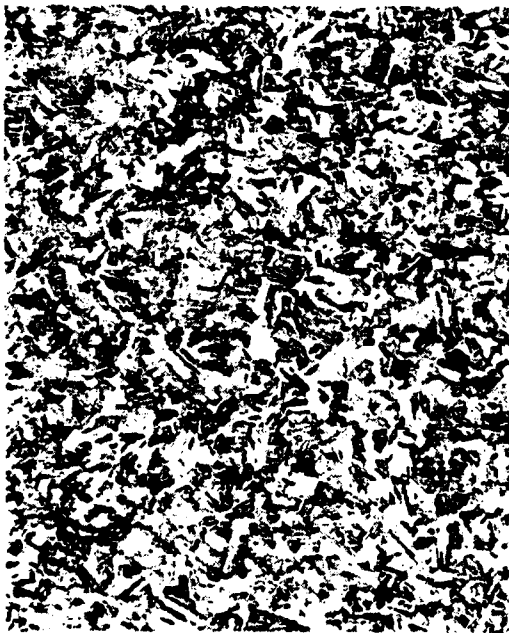
^a Weight PercentTable 2. Mechanical Properties of PH Stainless Steel Alloys.^a

Alloy	Condition	Hardness, R _c	σ_{ys} , MPa	σ_{ult} , MPa	RA, ^a %
17-4PH	"A"	35.5	983	1021	66
	H900	44.9	1396	1412	38
	H950	39.1	1183	1194	61
	H1000	38.7	1128	1137	61
	H1050	36.0	1066	1070	62
15-5PH	H1000	39.2	1018	1087	66
PH13-8Mo	H1000	45.0	1492	1540	54

^a RA = reduction in area.



(a)



(b)



(c)

FIGURE 2. Microstructures of 17-4PH Stainless Steel Heat-Treated to Conditions (a) "A", (b) H1000, and (c) H1050. Etchant: Kalling's Reagent. 250X

As a reference point, specimens from each material and heat treatment condition were first tested in air. Selected specimens were also tested in three different aggressive environments: 3.5% NaCl (pH 7.3), 3.5% NaCl + HCl (pH 1.5), and 3.5% NaCl + HCl (pH 1.5) plus cathodic polarization by means of a galvanic couple. The galvanic couple was produced by wrapping the individual specimens with aluminum foil. A small piece of foil was inserted within the notch area to ensure a galvanic reaction at the crack tip. A few specimens were also cathodically charged at room temperature for 16 hours just prior to fracture toughness testing in order to introduce atomic hydrogen into the chevron-notched materials. The specimens were immersed in a 10% H_2SO_4 solution containing 10mg/liter of As_2O_3 which promoted atomic hydrogen absorption. A current density of 9ma/cm^2 was used.

After fracture toughness testing, selected fracture surfaces were characterized by scanning electron microscopy (SEM) techniques to define fracture mode.

RESULTS AND DISCUSSION

The results of the fracture toughness testing are presented and discussed in this section. The influence of aggressive environments on the fracture toughness of 17-4PH, 15-5PH, and PH13-8Mo stainless steels heat treated to various conditions is also discussed. The results are compared with those of other investigators and are explained in terms of fracture surface appearance as revealed by SEM fractography.

FRACTURE TOUGHNESS

As discussed previously, fracture toughness values were obtained through the use of the Fractometer[®] II test system. Detailed test methods are presented elsewhere.¹³ A summary of the fracture toughness values obtained in this investigation is presented in Table 3 as a function of material, heat treatment, and testing environment. The tabulated fracture toughness values are averages of measurements made on three or more specimens. Data scatter was found to be less than $\pm 4.5\text{MPa/m}$ for any given test condition. Also tabulated is the percent change in fracture toughness from the K_{Ic} value measured in air for each heat treated group.

¹³TerraTek, University Research Park. Data Analysis Methods for Short Rod and Short Bar Fracture Toughness Tests of Metallic Materials, by L. M. Barker. Salt Lake City, Utah, TerraTek, March 1980. (TerraTek Report TR 80-12, publication UNCLASSIFIED.)

Table 3. K_{Ic} Values Of PH Stainless Steel Alloys.

Material	Condition	Test Environment	Average Fracture Toughness (MPa/m)	% Change from K_{Ic} In Air
17-4PH	H900	Air	60.8	-
		NaCl + HCl	59.6	2.0
		NaCl + HCl + Al	53.5	7.1
		Cathodic Charge	50.2	17.4
	H950	Air	106.9	-
		NaCl	97.0	9.3
		NaCl + HCl + Al	96.4	9.8
		NaCl + HCl	93.7	12.3
	H1000	Air	128.4	-
		NaCl	118.1	8.0
		Cathodic Charge	116.6	9.2
		NaCl + HCl + Al	109.2	15.1
	H1050	Air	123.0	-
		NaCl + HCl + Al	101.6	17.4
	"A"	Air	70.1	-
		NaCl	62.7	10.6
		NaCl + HCl + Al	59.3	15.3
		Cathodic Charge	55.8	20.3
15-5PH	H1000	Air	153.8	-
		NaCl + HCl + Al	140.4	8.7
PH13-8Mo	H1000	Air	101.5	-
		NaCl + HCl + Al	94.6	6.8

In order to determine the reliability of the chevron-notched specimen technique, results from this investigation of tests performed in air were first compared with data determined by others.^{14,15} It was found that these tests using chevron-notched specimens yielded results differing by a maximum of about 15% from data obtained by the ASTM E399 test method, except for those specimens that did not conform to geometry specifications, i.e., $B \geq (K_{Ic}/\sigma_{ys})^2$. This 15% difference can be accounted for by possible variations in material (differences in processing, composition, grain size, etc.).

Few data points from this investigation can be directly compared with data from either Fujii⁵ or Capeletti,⁷ since neither used the same aging treatments. Some of the comparisons, then, must be made by interpolation. The following observations can be made on fracture toughness of 17-4PH stainless steel. In the H900 condition, aluminum coupled specimens from this study yielded a K_{Isc} value of 56.5MPa/m, while aluminum coupled specimens from Fujii's research yielded a K_{Isc} value of 59.6MPa/m, a difference of only 5.2%. Also, in the aluminum coupled testing environment Fujii's H1025 and H1075 specimens yielded K_{Isc} values of 97.9 and 113.7MPa/m, respectively. This study found that the 17-4PH stainless steel in the H1050 condition, tested in the aluminum coupled environment has a K_{Isc} value of 101.6MPa/m. This is also in agreement with Fujii since the latter value falls between the two values mentioned above. Any discrepancies between Fujii's K_{Isc} values and this research could be due to small differences in chemical composition or processing history.

In comparing the results from this study with data from Capeletti, few similarities can be found. One of Capeletti's specimen groups was 17-4PH stainless steel in the H950 condition. Fracture toughness tests in a 69.5MPa helium inert atmosphere yielded a K_{Ic} value of 97MPa/m. Comparable specimens from this investigation tested in air yielded a K_{Ic} value of 106.9MPa/m, a difference of 9.3%. Although this shows fair agreement, the remainder of Capeletti's data contrast markedly with the data of this investigation and Fujii's data. This may be partly due to differences in testing environment, testing procedure, and/or chemical composition.

¹⁴DOD Aerospace Structural Metals Handbook, Vol. 2, published by Belfour Stulen, Inc., Traverse City, Michigan, 1980.

¹⁵J. E. Campbell, W. W. Gerberich, and J. H. Underwood. Application of Fracture Mechanics, American Society for Metals. Metals Park, Ohio, ASM, 1982, pp. 152-54.

As an example of how the testing environments affect fracture toughness, Figure 3 graphically shows the fracture toughness of 17-4PH stainless steel in the H900 condition as a function of testing environment. Again, as the testing environment becomes more severe, the fracture toughness of the material is decreased. Similarly, Figure 4 compares the fracture toughness of the various heat-treated conditions of 17-4PH, 15-5PH, and PH13-8Mo stainless steels tested in air and 3.5% NaCl + HCl + Al (coupled). Note that as the aging temperature is increased (lower yield strength), the fracture toughness increases both in air and in the aggressive environment.

MICROSCOPY

SEM fractography was employed to characterize the fracture behavior of the materials. The three materials tested, 17-4PH, PH13-8Mo, and 15-5PH stainless steels in all heat-treated conditions, exhibited a mixture of ductile fracture with quasi-cleavage for fracture toughness testing in air. Figure 5 illustrates the fracture surface at various magnifications of 17-4PH steel in the H900 condition. Figures 5(c) and 5(d) show the resulting quasi-cleavage. Figure 5(a) shows the point of the chevron-notch where fracture is initiated. Figure 6 illustrates how the fracture surface of 17-4PH steel changes from quasi-cleavage to dimpled rupture as the aging treatment was increased from the H900 to the H1050 condition. As the testing environment became more aggressive, the fracture mode changed from dimpled rupture/quasi-cleavage when testing in air to increasing amounts of cleavage when testing in increasingly aggressive environments. Figure 7 shows the fracture surfaces of 17-4PH steel in the H1000 condition evaluated in a variety of environments. It is observed that the sample tested in air experienced dimpled rupture, whereas, the specimen subjected to the galvanic environment (NaCl water, HCl, Al (coupled)) experienced pure cleavage, leading to a decreased fracture toughness.

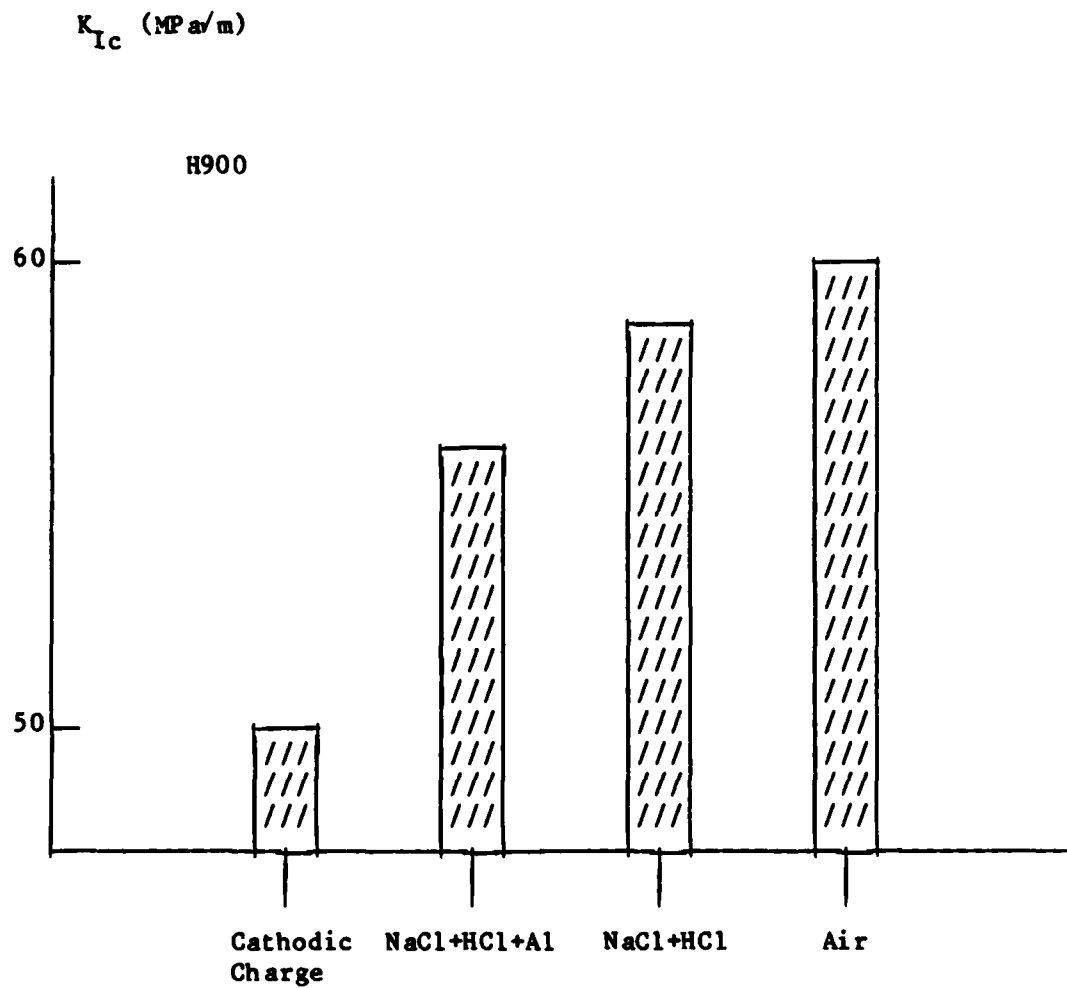


FIGURE 3. K_{Ic} Values of 17-4PH Stainless Steel (H900 Condition) in Various Environments.

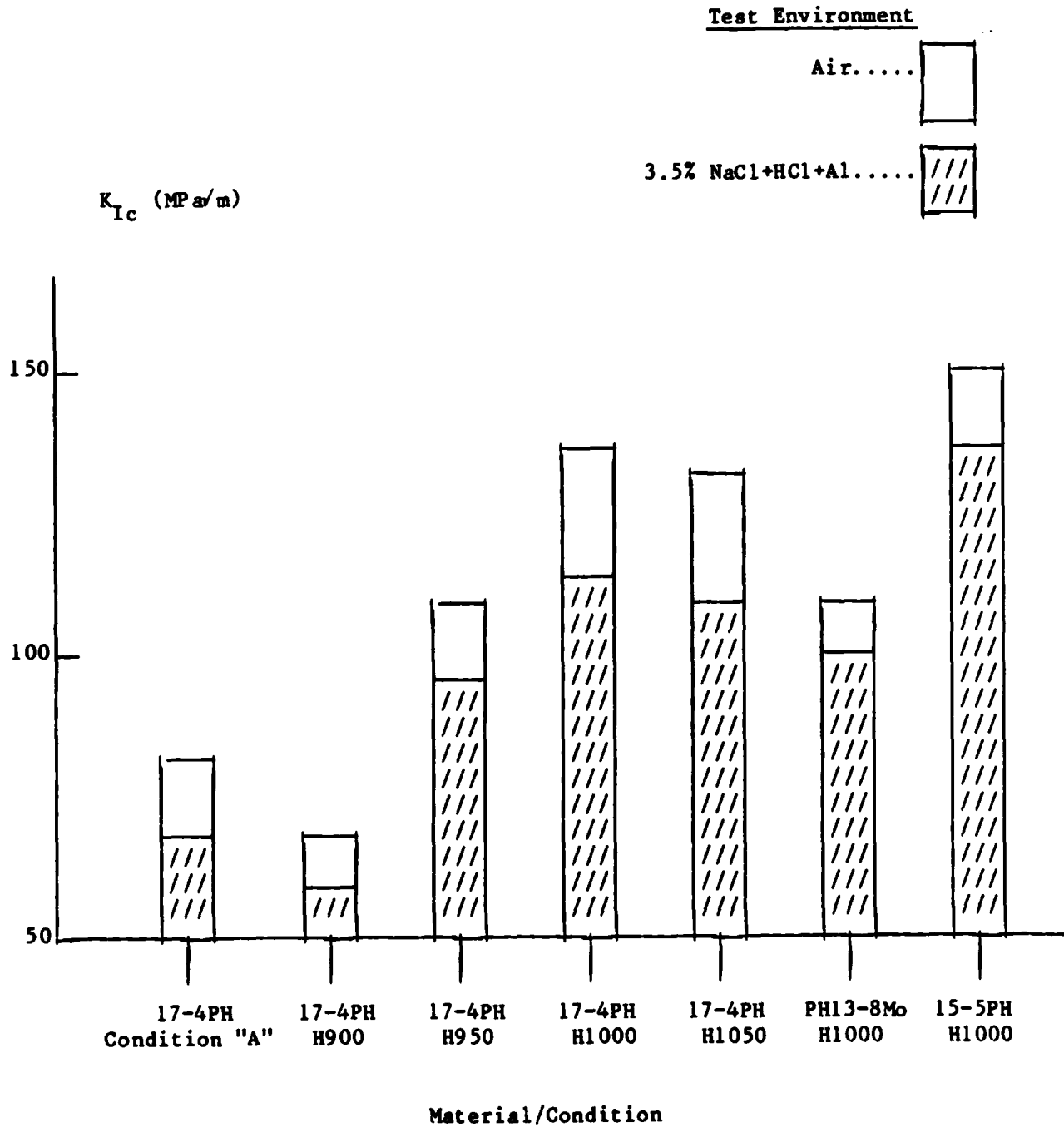
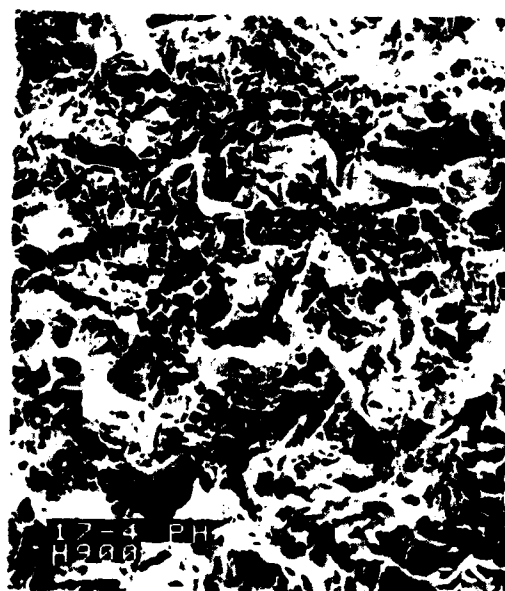


FIGURE 4. K_{Ic} Values of 17-4PH, 15-5PH, and PH13-8Mo (various conditions) Tested in Air and 3.5% NaCl+HCl+Al.



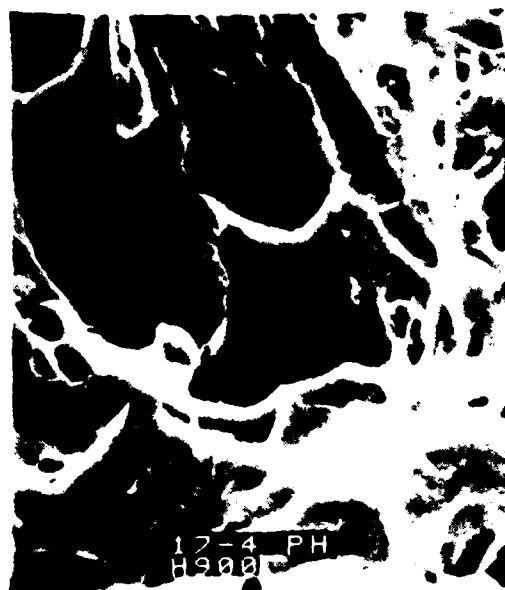
(a)



(b)



(c)



(d)

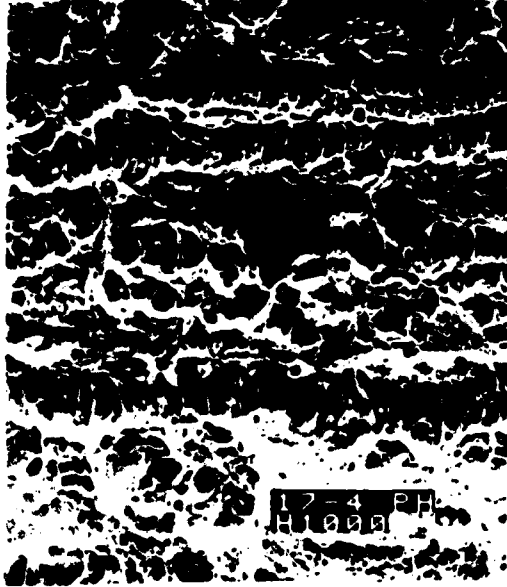
FIGURE 5. Fracture Surfaces of 17-4PH Stainless Steel Heat-Treated to the H900 Condition in Air, and Magnified to (a) 25X, (b) 250X, (c) 1500X, and (d) 3000X.



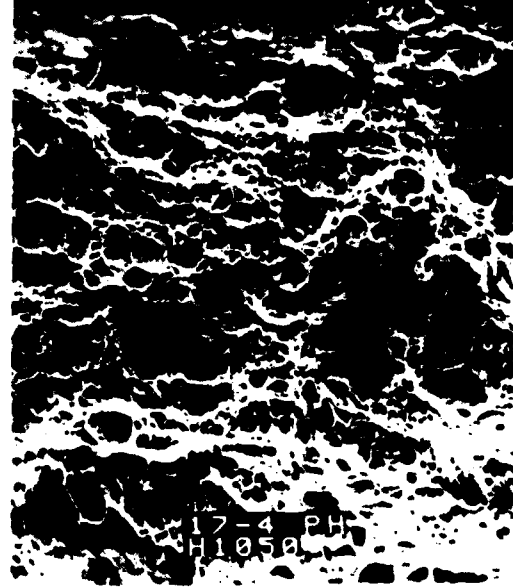
(a)



(b)

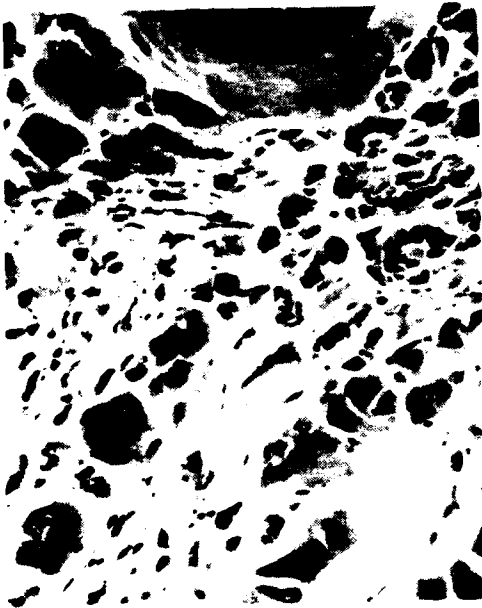


(c)



(d)

FIGURE 6. Fracture Surfaces of 17-4PH Stainless Steel Fracture Toughness Specimens Tested in Air in the (a) H900, (b) H950, (c) H1000, and (d) H1050 Conditions.



(a)



(b)



(c)

FIGURE 7. Fracture Surfaces of 17-4PH Stainless Steel Heat Treated to the H1000 Condition and Tested in (a) Air, (b) 3.5% NaCl, and (c) 3.5% NaCl + HCl + Al solution.

CONCLUSIONS

1. The chevron-notched test method yielded K_{Ic} data which substantiated K_{Ic} data obtained by the presently accepted ASTM E399 test method.
2. The chevron-notched test method can be used to evaluate K_{Iscc} of materials in aggressive environments, especially for quick comparative measurements.
3. Increasing yield strength decreased K_{Iscc} in all environments that were evaluated.
4. K_{Iscc} consistently decreased as the test environment became more aggressive. Cathodic polarization and cathodic charging leads to the greatest decrease in fracture toughness.
5. Results of the study indicate that hydrogen is the primary factor in promoting stress corrosion behavior in the PH stainless steels investigated.
6. As the fracture toughness decreases, due to aggressive testing environments, the fracture mode changes from dimpled rupture/quasi-cleavage to cleavage.

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